

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 348 (1968): French polish [CHD 20: Paints, Varnishes and Related Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



IS : 348 - 1968
(Reaffirmed 2009)

Indian Standard
SPECIFICATION FOR
FRENCH POLISH
(*First Revision*)

Fifth Reprint JUNE 1991
(Incorporating Amendment No. 1)

UDC 667.82

© Copyright 1977

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

IS : 348 - 1968
(Reaffirmed 1976)

Indian Standard

SPECIFICATION FOR FRENCH POLISH

(First Revision)

Paints and Allied Products Sectional Committee, CDC 8

Chairman

SHRI W. E. NORRIS

Representing

Goodlass Nerolac Paints Ltd, Bomb

Members

SHRI H. N. RAMACHAR (<i>Alternate to</i> Shri W. E. Norris)	
DR P. K. BHANDARI	Shalimar Paints Ltd, Calcutta
SHRI K. K. CHOWDHRY	Ministry of Defence (R & D)
SHRI I. D. SINGH (<i>Alternate</i>)	
SHRI B. K. DAS GUPTA	Ministry of Defence (R & D)
SHRI I. D. SINGH (<i>Alternate</i>)	
SHRI R. C. DAS GUPTA	National Test House, Calcutta
SHRI S. K. BOSE (<i>Alternate</i>)	
DIRECTOR OF SCIENTIFIC RESEARCH (NAVY)	Naval Headquarters
SHRI K. S. GANESAN	The Hyderabad Allwyn Metal Works Ltd, Hyderabad
SHRI ISHWAR SINGH (<i>Alternate</i>)	
DR JOSEPH GEORGE	Central Building Research Institute (CSIR), Roorkee
SHRI G. W. KAPSE (<i>Alternate</i>)	
SHRI N. GOPALKRISHNAN	Indian Aluminium Co Ltd, Calcutta
SHRI A. K. CAPRIHAN (<i>Alternate</i>)	
JOINT DIRECTOR (M & C)	Railway Board (Ministry of Railways)
SHRI L. R. FIALHO (<i>Alternate</i>)	
SHRI T. K. S. MANI	Addisons Paints and Chemicals Ltd, Madras
SHRI M. B. SATYANARAYANA (<i>Alternate</i>)	
SHRI M. N. RAO	Indian Paint Association, Calcutta
SHRI B. V. DALAL (<i>Alternate</i>)	
SHRI R. K. ROKADE	The Premier Automobiles Ltd, Bombay
SHRI P. K. DESHPANDE (<i>Alternate</i>)	
SHRI N. K. SEN	Development Commissioner, Small Scale Industries
SHRI P. R. MALHAN (<i>Alternate</i>)	
SHRI K. N. R. SHARMA	Directorate General of Technical Development
SHRI A. SINHA	Indian Paint Research Association, Calcutta
SHRI N. B. SENGUPTA (<i>Alternate</i>)	
DR M. A. SIVASAMBAN	Regional Research Laboratory, Hyderabad

(Continued on page 2)

BUREAU OF INDIAN STANDARDS

MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

IS : 348 - 1968

(Continued from page 1)

<i>Members</i>	<i>Representing</i>
SUPERINTENDING SURVEYOR OF WORKS (I)	Central Public Works Department, New Delhi
SURVEYOR OF WORKS (I) (<i>Alternate</i>)	
SHRI Y. S. SWAMY	L.C.I. (India) Private Ltd, Calcutta
SHRI H. J. MISTRY (<i>Alternate</i>)	
SHRI D. DAS GUPTA, Director (Chem)	Director General, ISI (<i>Ex-officio Member</i>)
<i>Secretaries</i>	
SHRI M. S. SAXENA	
Deputy Director (Chem), ISI	
SHRI S. ARAVAMUDHAN	
Assistant Director (Chem), ISI	

Finished Products Subcommittee, CDC 8 : 6

<i>Convener</i>	
SHRI T. K. S. MANI	Addiaons Paints and Chemicals Ltd, Madras
<i>Members</i>	
SHRI P. K. ADHIKARI	Jenson and Nicholson (India) Ltd, Calcutta
SHRI N. S. BHARATIA	Indian Paint Association, Galcutta
DR P. K. BHANDARI (<i>Alternate</i>)	
SHRI K. K. CHOWDHRY	Ministry of Defence (R & D)
SHRI B. K. DAS GUPTA (<i>Alternate</i>)	
SHRI R. C. DAS GUPTA	National Test House, Calcutta
JOINT DIRECTOR (M & C)	Railway Board (Ministry of Railways)
SHRI V. L. MEHENDALE	Ministry of Defence (DGI)
SHRI M. N. RAO	Indian Paint Association, Calcutta
SHRI H. N. RAMACHAR (<i>Alternate</i>)	
DR M. A. SIVASAMBAN	Regional Research Laboratory (CSIR), Hyderabad

Subcommittee for Revision of Indian Standards on Finished Products, CDC 8: 13

<i>Convener</i>	
SHRI R. C. DAS GUPTA	National Test House, Calcutta
<i>Members</i>	
SHRI P. K. ADHIKARI	Indian Paint Association, Calcutta
SHRI O. L. GUPTA	Ministry of Defence (DGI)
JOINT DIRECTOR (M & C)	Railway Board (Ministry of Railways)
SHRI S. K. KHASNOBIS	Hindustan Motors Ltd, Calcutta
SHRI M. N. RAO	Indian Paint Association, Calcutta

Indian Standard

SPECIFICATION FOR FRENCH POLISH (*First Revision*)

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 17 October 1968, after the draft finalized by the Paints and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1952 and was based largely on the *interim* co-ordinated draft, compiled by the Co-ordinating Subcommittee of the No. 5 Standing Committee on Specifications for Paints and Allied Stores of the General Headquarters, India (now Army Headquarters). Through use of the standard, technical comments were received from important users that the composition of the material based on shellac and denatured spirit only did not result in good performance. Taking cognizance of these comments, the composition clause has been suitably modified to permit the use of other alcohol soluble ingredients as may be considered necessary to improve the performance of the material. To safeguard the use of resins, other than rosin, a limit on iodine value of the residue, obtained after evaporation of the polish has been stipulated. A new specification limit for matter insoluble in hot alcohol has also been added.

0.2.1 The concerned Committee had, however, felt that the material is not intended for use on bare wood. A coat of liquid wood filler (*see* IS: 345-1952*) should normally be applied and rubbed out. French polish is then applied as a finishing coat to obtain a shiny, smooth and glossy finish.

0.2.2 Apart from the above, this revision has been aligned with the latest revised versions of other standards in the series. Metricization, already accomplished through Amendment No. 1 (February 1962), has been incorporated in the body of this standard. This revised standard, it is hoped, would bring it up to date in consonance with the needs of the users of this material.

0.2.3 The availability of denatured spirit in the country had been uncertain due to various reasons. The concerned technical committee, therefore, decided to permit the use of isopropyl alcohol as an alternative to denatured spirit through Amendment No. 1 of this standard.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing

Specification for wood filler, transparent, liquid.

IS : 348 - 1968

the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for the material commercially known as french polish. The material is used for finishing woodwork.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 1303-1963† and under **2** of IS : 197-1952‡ shall apply.

3. REQUIREMENTS

3.1 Description — The material shall be free from visible impurities.

3.2 Composition — The material shall contain 25 ± 1 percent by weight of shellac (conforming to IS: 16-1956§) dissolved in denatured spirit (conforming to IS: 324-1959||) or isopropyl alcohol or a mixture of the two together with other alcohol soluble ingredients as may be necessary so as to improve the performance of the material.

3.3 The material shall also comply with the requirements given in Table 1.

4. PACKING AND MARKING

4.1 Packing — The material shall be suitably packed in stoneware or glass containers or as agreed to between the purchaser and the supplier. The packing is subject to the provisions of the law in force in the country at that time.

4.2 Marking — Each container shall be marked with the following:

- a) Name of the material;
- b) Name of the manufacturer and/or trade-mark, if any;
- c) Volume of the material; and
- d) Month and year of manufacture.

* Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to paints (*revised*).

‡Methods of test for varnishes and lacquers. (Since revised).

§Specification for shellac (*revised*).

||Specification for ordinary denatured spirit (*revised*).

TABLE 1 REQUIREMENTS FOR FRENCH POLISH

(Clause 3.3)

SL NO.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST REF TO	
			Appendix	CI No. in IS : 197-1952*
(1)	(2)	(3)	(4)	(5)
i)	Drying time, hard dry	Not more than 2 hours	—	7.2
ii)	Finish	Smooth and glossy	—	8
iii)	Ash content, percent by weight, <i>Max</i>	0.50	—	15
iv)	Iodine value of residue†, <i>Max</i>	20.0	A	—
v)	Colour index	Shall not exceed 18.0	B	—
vi)	Matter insoluble in hot alcohol of the residue, percent by weight, <i>Max</i>	0.5	C	—
vii)	Keeping properties	Not less than one year from the date of manufacture	—	10

*Methods of test for varnishes and lacquers.

†Obtained after evaporation of the material at 40°C under vacuum (*see* Note under A-4.1).

4.2.1 Each container shall have a caution label 'FLAMMABLE' together with the corresponding symbol for labelling of dangerous goods. (*see* Fig. 3 of IS: 1260-1958*).

NOTE 1 — Necessary safeguards against the risk arising from the storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all times to prevent accidents by fire or explosion.

NOTE 2 — Except when they are opened for the purpose of cleaning and rendering them free from vapours, all empty tanks or other containers shall be kept securely closed.

4.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

*Code of symbols for labelling of dangerous goods. (Since revised and split into various parts).

IS : 348 - 1968

5. SAMPLING

5.1 Representative samples of the material shall be prepared as prescribed under 3 of IS : 197-1952*.

6. TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed in IS : 197-1952* and Appendices A to C of this standard. References to relevant clauses of IS : 197-1952* are given in col 5 and to appendices in col 4 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table 1, Sl No. (iv)*]

DETERMINATION OF IODINE VALUE OF RESIDUE

A-0. GENERAL

A-0.1 Outline of the Method — The material is evaporated at 40°C under vacuum and the iodine value of the residue determined by Langmuir method.

A-1. APPARATUS

A-1.1 Thermometer — of range 0° to 100°C with a smallest division of 0.5° conforming to schedule 7 of IS: 2480-1964‡.

A.2. REAGENTS

A-2.1 Standard Shellac — With every set of tests, run a determination on a sample of unadulterated shellac, the iodine value of which is known and has been certified by a recognized laboratory. The iodine number obtained for the standard shellac shall be within ± 0.5 of the known iodine number.

*Methods of test for varnishes and lacquers.

†Specification for water, distilled quality (*revised*).

‡Specification for general purpose glass thermometers, (Since revised).

A-2.2 Acetic Acid — pure grade conforming to IS : 695-1967*.

A-2.3 Chloroform — acetone-frec.

A-2.4 Standard Sodium Thiosulphate Solution — 0.1 N approximately (see 32 of IS:2316-1968†).

A-2.5 Starch Solution — Make a paste of 0.2 g of soluble (potato) starch in cold water and pour into 100 ml of boiling water. Boil for 5 minutes, cool and bottle. The solution shall be prepared freshly every 2 or 3 days.

A-2.6 Potassium Iodide Solution — Prepare fresh solution by dissolving 10 g of potassium iodide, free from potassium iodate, in 90 ml of water.

A-2.7 Wijs Iodine Monochloride Solution — Dissolve 13 g of iodine in a litre of the acetic acid, using gentle heat, if necessary, and determine the strength by titration with standard sodium thiosulphate solution. Set aside 50 to 100 ml of the solution and introduce dry chlorine gas into the remainder until the characteristic colour change occurs, and the halogen content is nearly doubled, as ascertained again by titration. If the halogen content has been more than doubled, reduce it by adding the requisite quantity of the iodine-acetic acid solution. A slight excess of iodine does not harm, but avoid an excess of chlorine.

Example — If the titration of 20 ml of original iodine-acetic acid solution required 22 ml of standard thiosulphate, 20 ml of the finished Wijs solution should require between 43 and 44 ml, and not more than 44 ml, of the same sodium thiosulphate solution.

A-3. PROCEDURE

A-3.1 Take the required amount of the material in a porcelain dish and evaporate the volatile matter at 40°C under vacuum. Cool the residue obtained to room temperature and preserve this for the determination of iodine value and matter insoluble in hot alcohol (see Appendix C).

A-3.1.1 Accurately weigh about 0.2 g of the residue prepared as given in **A-3.1** (see Note under **A-3.1.2**) and introduce into a 250-ml clean, dry, glass bottle having a ground-glass stopper. Add 20 ml of acetic acid and place the bottle on the top of a hot water-bath at 65 to 70°C, swirling the bottle occasionally, until solution is complete, except for the wax. This shall not require more than 15 minutes. Unadulterated shellac dissolves with some difficulty; solution is quicker according to the percentage of rosin present. Add 10 ml of chloroform and cool the solution to room temperature. Before adding the Wijs solution, allow the bottle to stand at room

*Specification for acetic acid (revised).

†Methods of preparation of standard solutions for colorimetric and volumetric analysis (first revision).

temperature for at least 30 minutes. Add 20 ml of the Wijs solution which shall also be at room temperature, from a pipette having a rather small delivery aperture (about 30 seconds). Close the bottle and note the time. Keep the bottle at room temperature for one hour that the shellac is in contact with the Wijs solution. Swirl the bottle occasionally during the one hour. After exactly one hour, add 10 ml of potassium iodide solution, washing into the bottle with it any Wijs solution left on the stopper. Titrate the solution immediately. Run in rapidly 25 to 30 ml of the standard sodium thiosulphate solution and shake vigorously until the solution assumes a straw colour. Now add 15 ml of starch solution and slowly finish the titration. The endpoint is sharp. Disregard any colour returning after about 30 seconds.

A-3.1.1.1 If a number of samples are being run, at least 5 minutes shall be allowed between the additions of the Wijs solution to facilitate titrations.

A-3.1.2 A blank determination shall be run under the same conditions, on the same reagents and by the same person but without using the material.

NOTE — In wetting residue (consisting mainly of shellac) some difficulty is experienced at times on account of its electrical properties. In very dry weather, handling of the material may electrify it. It may, therefore, be necessary to leave it in the balance pan at rest for a few minutes before determining the weight.

A-4. CALCULATION

A-4.1 Calculate the iodine value of the residue as follows:

$$\text{Iodine value} = \frac{12.69 (V_2 - V_1) N}{W}$$

where

V_2 = 33 volume in ml of standard sodium thiosulphate solution required for blank;

V_1 = volume in ml of standard sodium thiosulphate solution required for material;

N = normality of the standard sodium thiosulphate solution, and

W = weight in g of the material taken for test.

NOTE — The requirement for this characteristic (*see* Table 1) has been fixed as 20 since the iodine value of shellac has been arbitrarily taken as 16.5. A little increase over 16.5 in the iodine value of residue should not be taken as adulteration with rosin. By stipulating the iodine value as 20, allowance has been made to take care of the higher iodine value of resins other than rosin that may have been added to the material to the extent of 3 percent to improve its gloss and performance.

APPENDIX B

[Table 1, Item (v)]

DETERMINATION OF COLOUR INDEX

B-0. GENERAL

B-0.1 Outline of the Method — The colour index is determined by comparing the colour of 0.005 N solution of iodine (in potassium iodide solution) with the material by diluting the latter progressively with alcohol until a close match is obtained.

B.1. REAGENTS

B-1.1 Alcohol — 95 percent (by volume) rectified spirit (*see* IS : 323-1959*) or 95 percent (by volume) denatured spirit (*see* IS : 324-1959†) provided that it is colourless.

B-1.2 Standard Iodine Solution — Prepare the standard 0.005 N solution of iodine by introducing 5 ml of 0.1 N iodine solution (*see* 24 of IS : 2316-1968‡), with a burette, into a measuring flask and making up to 100 ml with water. The solution corresponds to colour index 5. Shake the solution before use.

B-1.3 Test Solution — Weigh accurately a quantity of the material equivalent to 10.0 g of dry shellac and dilute to 100 ml with ethyl alcohol (95 percent by volume). Filter the solution in an ordinary funnel using a medium grade filter paper. Discard the first 15 ml of the filtrate and then collect the clean filtrate for the test.

B.2. PROCEDURE

B-2.1 Transfer 5 ml of the filtered test solution prepared as in **B-1.3** to a thin-walled test-tube, by means of a pipette. Take an adequate volume of the standard iodine solution in another test tube similar in every respect for matching. Compare the colour of the two solutions holding the test-tubes against light with a piece of moistened filter paper or opal glass interposed in between the light source and the test-tube. Add alcohol from a burette to the test solution with shaking until the colour is the same as that of the standard solution. Note the volume of alcohol added.

B-2.1.1 It will be found advantageous to use a standard type of light source and a viewing cabinet to cut off extraneous light.

*Specification for rectified spirit (*revised*).

†Specification for ordinary denatured spirit (*revised*).

‡Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

IS : 348 - 1968

B-3. CALCULATION

B-3.1 The volume in millilitres or alcohol so added plus five, or the total volume in millilitres of the test solution after such dilution, is the colour index of the sample.

B-4. MODIFIED METHOD FOR LIGHT COLOURED MATERIAL

B-4.1 For samples having a colour index of less than 5, start with 0.001 N iodine solution and 10 ml of test solution prepared as in **B-1.3**. Follow the same procedure as above. The colour index shall be the final volume to which the test solution has to be diluted for matching the 0.001 N iodine solution, divided by 10.

APPENDIX C

[*Table 1, Item (vi)*]

DETERMINATION OF MATTER INSOLUBLE IN HOT ALCOHOL

C-0. GENERAL

C-0.1 The matter insoluble in hot alcohol is determined by extracting a known weight of non-volatile portion of the material with 95 percent (by volume) ethyl alcohol and determining the percentage of the undissolved residue by either (as may be agreed) of the two methods described under **C-1** and **C-2**.

C-1. METHOD I

C-1.1 Apparatus — The apparatus shall consist of the following.

C-1.1.1 Condenser — all glass, of the type and dimensions shown in Fig. 1, with the tip cut at an angle of 45 degrees. It shall have two holes at the tip to fasten the siphon tube.

C-1.1.2 Siphon Tube — of glass, of the type and dimensions shown in Fig. 1. The siphon tube shall have 2 holes near the top for a wire to be tube fastened to the condenser tip, leaving about 6 mm space between the top of the tube and the condenser tip.

C-1.1.3 Conical Flask — heat resistant, wide mouthed, preferably of borosilicate glass, 176 ± 3 mm in height and 48 ± 2 mm in inside diameter at the top. The flask shall have a tight fitting cork, 25 mm in depth and bored to fit the stem of the condenser. The bottom of the cork shall be just above the holes for the wire in the condenser. To support the flask, a suitable ring support with iron clamp and nichrome or iron gauze shall be used. The gauze shall not have an asbestos covering.

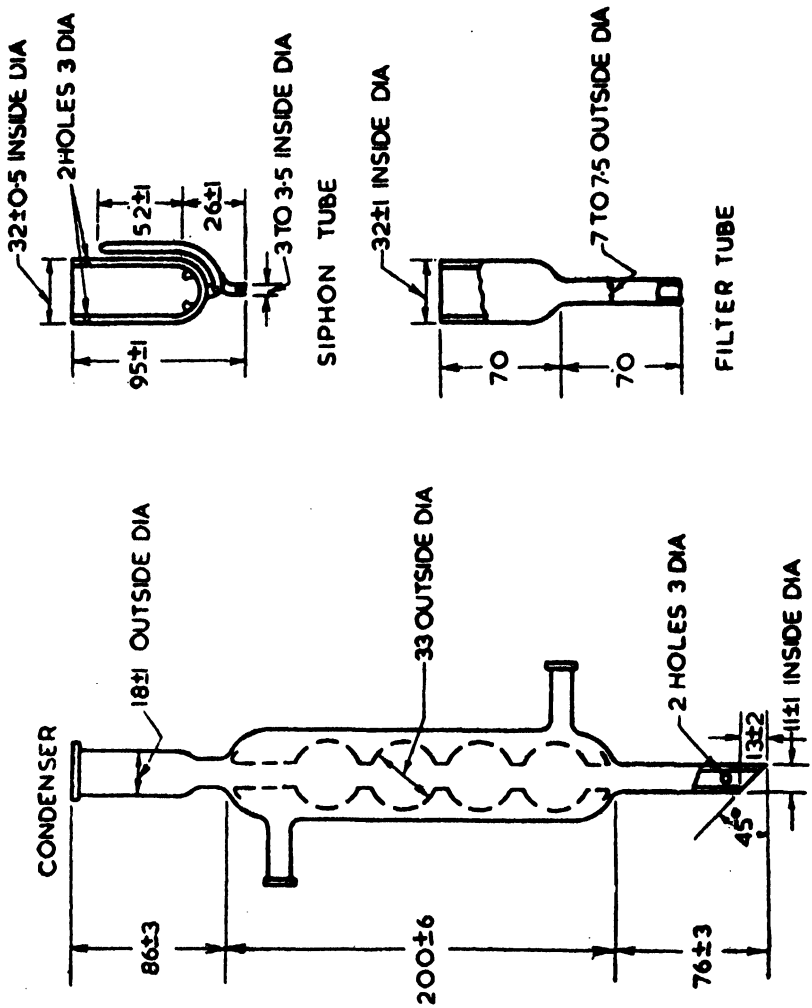


FIG. 1 EXTRACTION APPARATUS FOR DETERMINING MATTER INSOLUBLE IN HOT ALCOHOL, METHOD I

C-14.4 Filter Tube — a carbon filter tube of the type and dimensions shown in Fig. 1, with a light spiral spring at the bottom to hold up the extraction cartridge. The stem of the filter tube shall be fitted with a rubber stopper and firmly held in a hot water-bath.

C-1.1.5 Extraction Cartridge — *fat-free paper extraction cartridge 26 ± 1 mm in diameter and 60 ± 1 mm in height.

C-1.1.6 Weighing-Bottle — glass stoppered, 80 ± 1 mm in height and 40 ± 1 mm in diameter.

C-1.1.7 Hot Water-Bath — suitable one as shown in Fig. 2. The cover shall have a flanged hole, for a 200 ml beaker, and also a hole, through which the top of the filter tube projects. Directly below this hole, in the bottom of the bath, shall be a flanged hole to hold the rubber stopper through which the stem of the filter tube extends, to discharge into the bottle or flask. The hot water bath shall be mounted on a low tripod or stand.

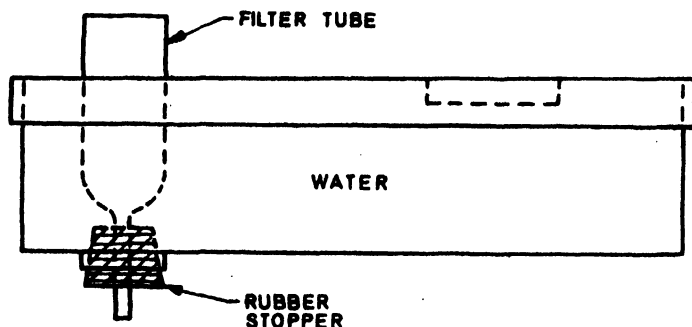


FIG. 2 HOT WATER-BATH FOR DETERMINING MATTER INSOLUBLE IN HOT ALCOHOL, METHOD I

C-1.1.8 Gas Burner — low form, adjustable, Bunsen type, carrying a draught shield. Any other suitable heating device may be employed.

C-1.2 Reagent — The following reagent is required.

C-1.2.1 Ethyl Alcohol — 95 percent (by volume) rectified spirit (see IS: 323-1959[†]); or 95 percent (by volume) denatured spirit (see IS: 324.1959[‡]).

C-1.3 Preparation of Extraction Cartridge — Place 125 ml of the alcohol in the flask and a cartridge in the siphon tube. Introduce the

*Schleicher and Schull No. 603 or its equivalent is suitable.

[†]Specification for rectified spirit (revised).

[‡]Specification for ordinary denatured spirit (revised).

siphon tube into the flask and connect it to the condenser making sure that there is an ample flow of cold water through the condenser. Adjust the flame of the burner so as to give a cycle of filling and emptying in the siphon tube every 2 minutes, and extract for 30 minutes. Dry the cartridge in an oven at a temperature not exceeding 105°C. At the end of 3 hours, weigh it in a tared weighing bottle which has been kept in a desiccator over sulphuric acid, lifting the stopper of the bottle momentarily before weighing. Continue drying, and weigh as before, after each hour of drying, until the loss in weight between successive weighings does not exceed 2 mg.

C.1.3.1 For referee tests new cartridges shall be used. A number of cartridges may be extracted, dried, weighed and kept in weighing bottles in a desiccator until needed for use.

C-1.4 Procedure

C-1.4.1 Weigh 4.5 to 5.5 g of the sample prepared as prescribed in **A-3.1** to an accuracy of 0.01 g, place in a 200-ml, tall, lipped beaker, add 125 ml of alcohol, stir with a glass rod, cover with a watch-glass, and place in the hot water-bath (*see* Fig. 2). Boil the solution vigorously for 30 minutes to ensure complete solution of the material. Keep the volume of alcohol constant.

C-1.4.2 Meanwhile place an extracted and weighed cartridge in the filter tube. Maintain the hot water around the tube at not less than 90°C. Wet the cartridge with hot alcohol, and decant the boiling solution into the heated cartridge until the beaker is nearly empty.

C-1.4.3 Wash the remaining solution and the insoluble matter into the cartridge, using a 'policeman' if necessary, with successive portions of hot alcohol contained in a wash bottle kept hot on the water-bath. Finally, wash the cartridge from the top downwards with a fine stream of hot alcohol. A complete washing and transfer from the original beaker will require at least 75 ml of hot alcohol.

C-1.4.4 Transfer the cartridge containing the insoluble matter to the extraction apparatus, place 125 ml of alcohol in the extraction flask and connect up the apparatus. Start the water flowing through the condenser, making sure that there is an adequate supply for efficient condensation. Light the burner and time the extraction from the first emptying of the siphon, running the extraction for exactly one hour. Adjust the Bunsen burner so that a complete filling and emptying of the siphon tube takes place every 2 minutes, as determined by a stop-watch, or preferably by a good two-minute sand glass, one for each extraction apparatus.

C-1.4.4.1 In this way, exactly 30 cycles per hour are accomplished. If this cycle is not meticulously maintained, neither check results on duplicate samples in the same laboratory, nor concordant figures from one laboratory

to another can be obtained, even when working on the same sample. It is also necessary to guard the apparatus from draughts while in operation; otherwise the proper cycle rate cannot be maintained.

C-1.4.4.2 Occasionally, residues are encountered which do not yield the required number of 30 siphonings per hour, due to slow filtration. In these instances, continue the extraction until 30 siphonings have been accomplished or repeat the test with a 2-g sample.

C-1.4.5 Remove the cartridge, drain in an upright position on filter paper and dry in an oven at $100 \pm 2^\circ\text{C}$. After drying for 2 hours, place it in the weighing bottle, cool in a desiccator over sulphuric acid, and weigh, removing the stopper momentarily just before weighing. Repeat drying and weighing as before, after each hour of drying, until the loss in weight between successive weighings does not exceed 2 mg. From the weight of the residue and the weight of the sample, calculate the percentage of insoluble matter.

C-1.5 Calculation

$$\begin{array}{l} \text{Matter insoluble in hot alcohol,} \\ \text{percent by weight} \end{array} = \frac{100 w}{W}$$

where

w = weight in g of residue, and

W = weight in g of sample taken.

C-2. METHOD II

C-2.1 Apparatus — The apparatus shall consist of the following.

C-2.1.1 **Siphon Tube* — of glass, of the Knoefler type having minimum internal dimensions of 52 mm height and 32 mm diameter, resting in an adaptor tube, in such a way that the siphon tube is surrounded by the ascending vapours of the boiling solvent (see Fig. 3).

C-2.1.2 *Condenser* — of any convenient pattern.

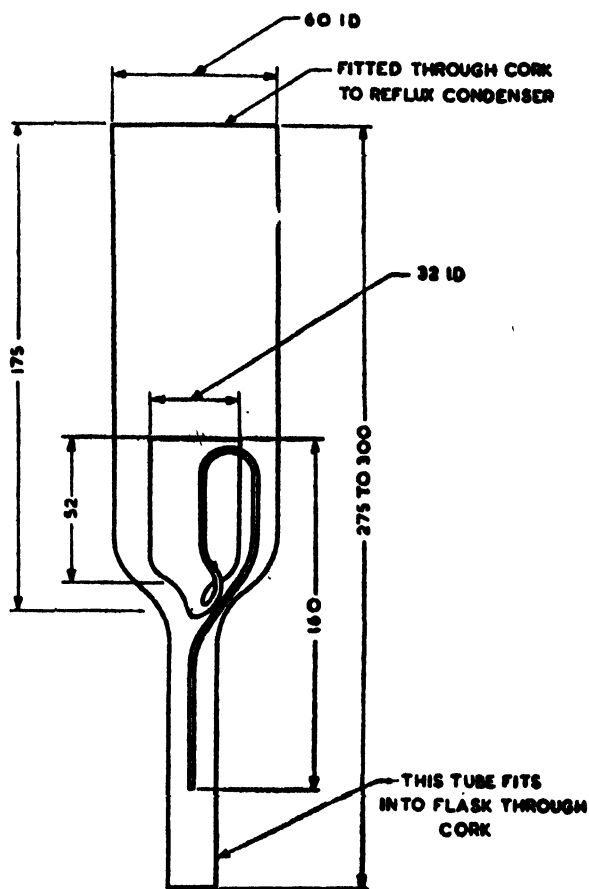
C-2.1.3 *Flask* — of suitable size.

C-2.1.4 †*Filter Paper* — 12.5 cm diameter, medium grade.

C-2.1.5 *Weighing Bottles* — of glass, height 80 ± 1 mm, diameter 40 ± 1 mm, with ground-glass stoppers.

*The type of extraction apparatus used is not critical, provided that it is of such a design as to ensure a continuous series of extractions at approximately the boiling temperature of the solvent. If preferred, the apparatus described for Method I, consisting of siphon tube, condenser and flask, could be satisfactorily used.

†Whatman No. 1 or its equivalent is suitable.



All dimensions in millimetres.

FIG. 3 SIPHON TUBE AND ADAPTOR

C-2.2 Assembly of Apparatus — The siphon tube, adaptor, condenser and flask shall be assembled with the aid of corks or ground glass joints so that the solvent can be kept boiling in the flask and its vapour passed upwards by way of the adaptor to the condenser. The refluxing solvent shall run from the condenser into the cup of the siphon tube.

C-2.3 Reagent — The following reagent is required.

C-2.3.1 *Ethyl Alcohol*—95 percent (by volume) rectified spirit (see IS : 323-1959*); or 95 percent (by volume) denatured spirit (see IS : 324-1959†).

C-2.4 Procedure

C-2.4.1 Fold a filter paper so that it forms a completely closed envelope, as illustrated in Fig. 4. Mark this paper *S* for sample; wrap it closely in a second filter paper marked *C* for counterpoise. Separate the filter papers and dry in an oven at $100 \pm 2^\circ\text{C}$ for 30 minutes. Rapidly transfer to weighing bottles which have been kept in a desiccator over concentrated sulphuric acid. Place each bottle with its contents back in the desiccator for 20 minutes, then weigh by counterpoise, preferably using a rapid weighing balance of the aperiodic type.

C-2.4.2 Weigh 4.5 to 5.5 g of the sample prepared as prescribed in **A-3.1** to an accuracy of 0.01 and place in the filter paper envelope *S*, and fold in the original folds taking care not to leave any channel through which finely divided material might afterwards escape. Again enclose in paper *C* and secure with thread. Place the resulting envelope in a 100 ml beaker and cover it with alcohol. Allow to stand overnight at room temperature. Transfer the envelope to the cup of the siphon tube and extract continuously with hot alcohol for 4 hours. Keep the envelope wholly below the surface of the alcohol when the cup is full. Maintain a rapid rate of extraction throughout, though the exact time taken for the cycle of filling and emptying the cup of the siphon tube is not critical.

NOTE — Double folds on the three sides as illustrated in (3) of Fig. 4 are recommended to ensure against inadvertent escape of finely divided material.

C-2.4.3 At the end of the specified time, remove the paper envelope, allow to drain, separate the two papers, dry each on a glass plate in the steam oven and then for 3 hours in a thermostatically controlled oven at $100 \pm 2^\circ\text{C}$. Place the papers rapidly in their respective weighing bottles, allow it to stand in the desiccator for 20 minutes and again weigh by counterpoise, after momentarily removing and replacing the stoppers in the usual manner. Dry the papers for a further period of one hour at $100 \pm 2^\circ\text{C}$ and weigh again; if there is a loss in weight in excess of 2 mg, repeat the processes of drying and weighing until the difference between successive weighings is less than 2 mg. Use the lowest weight in the calculation.

C-2.5 Calculation — as under C-1.5.

*Specification for rectified spirit (revised).

†Specification for ordinary denatured spirit (revised).

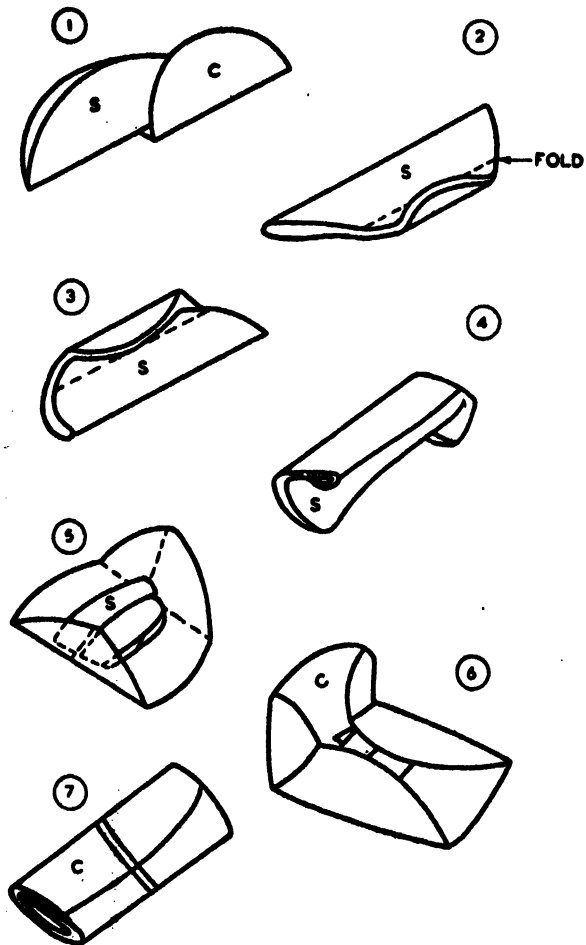


FIG. 4 FOLDING OF FILTER PAPER

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 331 01 31, 331

Telegrams: Manaksanstha
(Common to all Offices)

Regional Offices:

	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	{ 331 01 31 331 13 75
*Eastern : 1/14 C. I. T. Schema VII M, V. I. P. Road, Maniktola, CALCUTTA 700054	36 24 99
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	{ 2 18 43 3 16 41 41 24 42
Southern : C. I. T. Campus, MADRAS 600113	{ 41 25 19 41 29 16
†Western : Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093	6 32 92 95

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMADABAD 380001	{ 2 63 48 2 63 49
‡Peenya Industrial Area 1st Stage, Bangalore Tumkur Road BANGALORE 560058	{ 38 49 55 38 49 56
Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003	6 67 16
Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002	5 36 27
53/5. Ward No. 29, R.G. Barua Road, 5th Byelane, GUWAHATI 781003	3 31 77
5-8-56C L. N. Gupta Marg (Nampally Station Road), HYDERABAD 500001	23 10 83
R14 Yudhister Marg, C Scheme, JAIPUR 302005	{ 6 34 71 6 98 32
117/418 B Sarvodaya Nagar, KANPUR 208005	{ 21 68 76 21 82 92
Patliputra Industrial Estate, PATNA 800013	6 23 05
T.C. No. 14/1421, University P.O., Palayam TRIVANDRUM 695035	{ 6 21 04 6 21 17

Inspection Offices (With Sale Point):

Pushpanjali, First Floor, 205-A West High Court Road, Shankar Nagar Square, NAGPUR 440010	2 51 71
Institution of Engineers (India) Building, 1332 Shivaji Nagar,5 PUNE 411005	24 35

*Sales Office in Calcutta is at 5 Chowringhee Approach, P. O. Princep 27 68 00
Street, Calcutta 700072

‡Sales Office in Bombay is at Novelty Chambers, Grant Road, 89 65 28
Bombay 400007

‡Sales Office in Bangalore is at Unity Building, Narasimharaja Square, 22 36 71
Bangalore 560002